भारतीय मानक

Indian Standard

IS 4448 : 2022

बेंज़ोइक अम्ल, खाद्य ग्रेड — विशिष्टि

(दूसरा पुनरीक्षण)

Benzoic Acid, Food Grade — Specification

(Second Revision)

ICS 67.220.20

भारतीय मानक ब्यूरो
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December 2022 **Price Group 6**

Food Additives Sectional Committee, FAD 08

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

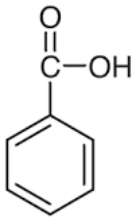
Food additives are added to improve the appearance, flavour, texture or storage properties of processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards have, therefore, been prepared by Bureau of Indian Standards to cover purity and identification of these substances. It is hoped that these standards would help in checking purity at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and national/international bodies.

Benzoic acid is bacteriostatic and bactericidal agent and also widely used as preservative for yeast and mould. It is widely used as antimicrobial agent.

Benzoic acid in free state is widely distributed in nature. Gum benzoic from Styrax benzoin, Acaroid resin from *Xanthorrhea*, scent glands of beaver, bark of wild black cherry tree, ripe cloves and oil of anise seed contain benzoic acid.

Benzoic acid is a permitted food additives as per Food Safety and Standards (Food Products Standards and Food Additives) Regulation, 2011. It is also included in food chemical codex and also falls under Indian Pharmacopoeia (IP), British Pharmacopoeia (BP) and United State Pharmacopoeia (USP).

Chemical Names and Formula — The recognized chemical names are benzoic acid, benzenecarboxylic acid and phenyl carboxylic acid. Empirical formula of benzoic acid is $C_7H_6O_2$. Its molecular weight is 122.12. Structural formula of benzoic acid is given below:



This standard was first published in 1967. It was first revised in 1994 considering requirements given in following International Standards as well as the practices being followed in the trade:

a) FAO Food and Nutrition Paper No. 4 — Specification for identity and purity of thickening agents, anticaking agents, antimicrobials, antioxidants and emulsifiers; published by the joint FAO/WHO Expert Committee on Food Additives;

b) Food Chemical Codex, 1981 Pub. National Academy of Sciences and National Research Council, Washington DC, USA; and

c) Council Directive 65/66/EEC of 26 January 1965 laying down specific criteria of purity for preservatives authorized for use in foodstuffs intended for human consumption.

In the first revision, solubility was brought under the description clause of the standard and was intended only as information regarding approximate solubility and not to be considered as a quality requirement. The requirement for polycyclic acid was deleted as this aspect was already covered under melting range. The requirement for lead was substituted by heavy metals and a requirement for loss on drying was added.

(*Continued on third cover*)

Indian Standard

BENZOIC ACID, FOOD GRADE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for benzoic acid for use as a food preservative.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.		*Title*
IS 1070 1992	:	Reagent grade water (*third revision*)
IS 1699 1995	:	Methods of sampling and test for food colours (*second revision*)

3 REQUIREMENTS

3.1 Description

Benzoic acid shall be in the form of white crystals, scales or needles. It shall have not more than a faint characteristic odour. Benzoic acid is slightly soluble in water and freely soluble in chloroform and 95 percent ethanol.

NOTE — The solubility is intended only for information regarding approximate solubility and is not to be considered as a quality requirement. It is of minor significance and should be considered as a means of identification or determination of purity and dependence must be placed on other specifications.

3.2 Identification Tests

3.2.1 *Reaction with Ferric Chloride*

Add 0.1 g of calcium carbonate and 5 ml of water to 0.1 g of the material. Heat gently and filter. To the filtrate, add 4.5 percent aqueous solution of ferric chloride. It shall form a buff-coloured precipitate.

3.2.2 Place a pinch of the sample in a dry test tube. Wrap the test tube about 4 cm from the bottom with moistened filter paper. Heat the test tube over a low flame. Benzoic acid shall sublime and crystals shall deposit in the colder part of the test tube leaving no residue at the bottom.

3.3 The material shall also conform to the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be filled in amber coloured glass containers, or any other well-closed containers, or suitable bag with inner lining of food grade material, with as little air space as possible. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Marking

4.2.1 Each container shall be legibly and indelibly marked with the following information:

a) Name of the material, including the words 'Food Grade';

b) Name of the manufacturer or his registered trade-mark, if any;

c) Net quantity when packed;

d) Lot/Batch No.;

e) Month and year of manufacture;

f) Best before ..... months from manufacture; and

g) Any other requirements as specified under the Legal Metrology (Packaged Commodities) Rules, 2011 and Food Safety and Standards (Packaging) Regulations, 2018 and Food Safety and Standards (Labelling and Display) Regulations, 2020.

Table 1 Requirements for Benzoic Acid
(*Clauses* 3.3 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Refer to	
			Annex of this Standard	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Purity (as $C_7H_6O_2$), percent by mass, on dry basis, *Min*	99.5	A	-
ii)	Melting range	121 ºC – 123 ºC	B	-
iii)	Sulphated ash, percent by mass, *Max*	0.05	C	-
iv)	Readily carbonizable substances	To conform to the test	D	-
v)	Readily oxidizable substances	To conform to the test	E	-
vi)	Loss on drying (for 3 hours over sulphuric acid or silica gel at ambient temperature in a desiccator) percent by mass, *Max*	0.5	-	-
vii)	Chlorinated organic compounds (as Cl_2), percent by mass, *Max*	0.07	F	-
viii)	Arsenic (as As), mg/kg, *Max*	3.0	-	**15** of IS 1699
ix)	Lead, mg/kg, *Max*	2.0	-	**15** of IS 1699

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provision of *Bureau of Indian Standards Act*, 2016 and Rules and Regulation framed there under and the product(s) may be marked with the Standard Mark.

5 SAMPLING

Representative samples of the material shall be drawn according to the method prescribed in **4** of IS 1699.

6 TESTS

6.1 Tests shall be carried out by the methods as specified in **3.2** and col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless otherwise, specified pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the experimental results.

ANNEX A
[*Table* 1, *Sl No.* i)]

DETERMINATION OF PURITY

A−1 REAGENTS

A−1.1 Phenol Red Solution

Warm 0.05 g of phenol red [phenolsulphonphthalein ($C_{19}H_{14}O_5S$)] with 2.85 ml of 0.05 N sodium hydroxide and 5 ml of 90 percent ethanol; after solution is effected, add a sufficient quantity of 20 percent ethanol to produce 250 ml.

A−1.2 Standard Sodium Hydroxide Solution — 0.5 N

A−1.3 Phenolphthalein Indicator

Dissolve 0.2 g of phenolphthalein ($C_{20}H_{14}O_4$) in 60 ml of 90 percent ethanol and add a sufficient quantity of water to produce 100 ml.

A−2 PROCEDURE

Weigh 2.5 g of benzoic acid to the accuracy of 0.1 mg which has been previously dried over concentrated sulphuric acid or over silica gel for 3 hours and dissolve in 15 ml of warm ethanol previously neutralized using phenol red. Add 20 ml of water and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

A−3 CALCULATION

A−3.1 Calculate the percentage of $C_7H_6O_2$ by the formula given below: Each ml of 0.5 N sodium hydroxide is equivalent to 0.061 06 g of $C_7H_6O_2$.

ANNEX B
[*Table* 1, *Sl No.* ii)]

DETERMINATION OF MELTING RANGE

B−1 APPARATUS

B−1.1 Capillary Tube

Thickness of wall 0.10 mm to 0.15 mm; internal diameter 0.9 mm to 1.1 mm.

B−1.2 Melting Point Apparatus

Provided with an appropriate liquid like paraffin or silicon oil and a stirring device, and fitted with an auxiliary thermometer.

B−2 PROCEDURE

B−2.1 Spread a small quantity of the finely powdered material in a thin layer and dry at a temperature below its melting temperature, or in a vacuum desiccator over sulphuric acid for 24 hours.

B−2.2 Transfer a quantity of the dried powder to a dry capillary tube and pack the powder by gently tapping the tube on a hard surface so as to form a tightly-packed column 2 mm to 4 mm high. Attach the capillary tube with its contents to a standard thermometer so that the closed end is at the level of the middle of the bulb, and heat in the melting point apparatus, regulating the rise of temperature during the first period to 3 °C per minute. When the temperature reaches to 116.5 °C the heating of the apparatus is adjusted, the rate of rise of temperature should be 1 °C to 2 °C per minute.

B−2.3 Take the reading of the temperature at which the material is observed to form droplets against the side of the tube, and of the temperature at which it is completely melted, as indicated by the formation of a definite meniscus.

B−2.4 To the temperature reading, apply the emergent stem correction, as follows:

Before starting the determination of the melting range, attach the auxiliary thermometer so that the bulb touches the standard thermometer at a point midway between the graduation for the expected melting range and the surface of the beating material. When the substance has melted, read the temperature on the auxiliary thermometer. Calculate the correction to be added to the temperature reading of the standard thermometer from the following formula:

$$0.000\ 16\ N\ (T - t)$$

where

N = The number of degrees of the scale of the standard thermometer between the surface

of the heating material and the level of the mercury;

T = The temperature reading of the standard thermometer; and

t = The temperature reading of the auxiliary thermometer.

B−3 INTERPRETATION OF RESULTS

B−3.1 The statement melting range, 121 °C to 123 °C means that the corrected temperature at which the material is observed to form droplets shall be at least 121 °C and that the material shall be completely melted at the corrected temperature, that is, 123 °C.

ANNEX C
[*Table* 1, *Sl No.* iii)]

DETERMINATION OF SULPHATED ASH

C−1 PROCEDURE

C−1.1 Weigh accurately 1.000 g to 2.000 g of the material in a tared crucible. Ignite until thoroughly charred. Cool, then moisten the residue with one ml of sulphuric acid, and cautiously ignite until the carbon is completely consumed. Conduct the ignition in a place protected from air currents, and use as low a temperature as possible to effect the combustion of the carbon. When the carbon has completely disappeared, cool the crucible in a desiccator and weigh. Note down the weight as ash.

ANNEX D
[*Table* 1, *Sl No.* iv)]

TESTFOR READILY CARBONIZABLE SUBSTANCES

D−1 REAGENTS

D−1.1 Sulphuric Acid — 94.5 to 95.5 percent

D−1.2 Matching Fluid

Composed of 0.2 ml of cobalt chloride solution (*see* **D−1.2.1**), 0.3 ml of ferric chloride solution (*see* **D−1.2.2**), 0.1 ml of cupric sulphate (*see* **D−1.2.3**) and 4.4 ml of water.

D−1.2.1 *Cobalt Chloride Solution*

Dissolve about 65 g or cobalt chloride ($CoCl_2.6H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place exactly 5 ml of this solution in a 25 ml iodine flask, add 5 ml of 3 percent hydrogen peroxide and 15 ml of 20 percent sodium hydroxide solution. Boil for 10 minutes, cool, and add 2 g of potassium iodide and 20 ml of 25 percent sulphuric acid. When the precipitate has dissolved, titrate the liberated iodine with 0.1 N sodium thiosulphate solution adding starch. Each ml of 0.1 N sodium thiosulphate is equivalent to 23.8 mg of $CoCl_2.6H_2O$. Adjust the final volume of the solution by adding enough of the mixture of hydrochloric acid and water to make such milliliter contain 59.5 mg of $CoCl_2.6H_2O$.

D−1.2.2 *Ferric Chloride Solution*

Dissolve 55 g of ferric chloride in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place 10 ml of this solution in a 250 ml iodine flask, add 15 ml of water and 3 g of potassium iodide and allow the mixture to stand for 15 minutes. Dilute with 100 ml of water, and titrate the liberated iodine with 0.1 N sodium thiosulphate solution, adding starch. Each milliliter of 0.1 N sodium thiosulphate is equivalent to 27.03 mg of $FeCl_3.6H_2O$. Adjust the final volume of the solution by addition of enough of the mixture of hydrochloric acid and water to make each ml contain 45.0 mg of $FeCl_3.6H_2O$.

D−1.2.3 *Cupric Sulphate Solution*

Dissolve about 65 g of cupric sulphate ($CuSO_4.5H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place 10.0 ml of this solution in a 250 ml iodine flask and add 40 ml of water, 4 ml of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1 N sodium thiosulphate adding starch. Each ml of 0.1 N sodium thiosulphate is equivalent to 24.97 mg of $CuSO_4.5H_2O$. Adjust the final volume of the

solution by addition of enough of the mixture of hydrochloric acid and water to make each ml contain 62.4 mg of $CuSO_4.5H_2O$.

D−2 PROCEDURE

D−2.1 Dissolve 0.500 g of benzoic acid in 5 ml of sulphuric acid; the solution shall not have deeper colour than the matching fluid.

ANNEX E
[*Table* 1, *Sl No.* v)]

TEST FOR READILY OXIDIZABLE SUBSTANCES

E−1 REAGENTS

E−1.1 Sulphuric Acid — 94.5 to 95.5 percent

E−1.2 Standard Potassium Permanganate Solution — 0.1 N

E−2 PROCEDURE

E−2.1 Add 1.5 ml of sulphuric acid to 100 ml of water, heat to boiling and add standard potassium permanganate solution in drops, until the pink colour persists for 30 seconds. Dissolve 1.000 g of benzoic acid in the heated solution, and titrate with standard potassium permanganate solution to a pink colour that persists for 30 seconds. The amount of 0.1 N standard potassium permanganate solution used shall not exceed 0.5 ml.

ANNEX F
[*Table* 1, *Sl No.* vi)]

TEST FOR CHLORINATED ORGANIC COMPOUNDS

F−1 REAGENTS

F−1.1 Sodium Hydroxide Solution — 0.1 N

F−1.2 Concentrated Nitric Acid — 69. 0 percent to 71.0 percent

F−1.3 Calcium Carbonate

F−1.4 Nitric Acid Dilute

Mix 190 ml of 50 percent nitric acid with sufficient water to make up 1 000 ml.

F−1.5 Silver Nitrate Solution — 0.1 N

F−1.6 Standard Hydrochloric Acid — 0.1 N

F−2 PROCEDURE

F−2.1 Dissolve 0.25 g of benzoic acid in 10 ml of sodium hydroxide solution. Acidify with concentrated nitric acid and filter off the precipitate. Mix the precipitate with 0.5 g of calcium carbonate, dry the mixture and then ignite. Take up the ignited residue in 20 ml of dilute nitric acid and filter. Mix the solution with 0.5 ml of silver nitrate solution. The turbidity shall be not more than that obtained in a similar volume by addition of 0.5 ml of silver nitrate solution and 0.05 ml of 0.1 N standard hydrochloric acid.

ANNEX G
(*Foreword*)

COMMITTEE COMPOSITION

Food Additives Sectional Committee, FAD 08

Organization	*Representative(s)*
CSIR-Indian Institute of Toxicology Research, Lucknow	DR YOGESHWAR SHUKLA (***Chairperson***)
All India Food Processors Association, (India)	MS SHREYA PANDEY SHRI KRISHNA KUMAR JOSHI (*Alternate*)
Association of Food Scientists and Technologists India, Mumbai	DR VIKAS SINGH CHAUHAN DR NANDINI P. SHETTY (*Alternate*)
Bose Institute, Kolkata	PROF GAOURISHNKAR
CSIR-Central Food Technological Research Institute, Mysore	DR NGASEPPAM IBOYIAMA SHRI ARUNA KUMAR (*Alternate*)
Confederation of Indian Food Trade and Industry, New Delhi	DR JASVIR SINGH MS PRIYANKA SHARMA (*Alternate)*
Confederation of Indian Industry, New Delhi	MS NEHA AGGARAWAL MS MAMTA ARORA BUDHIRAJA (*Alternate*)
Consumer Education and Research Centre, Ahmedabad	MS ANINDITA MEHTA MS DOLLY A. JANI (*Alternate*)
Consumer Guidance Society of India, Mumbai	DR SITARAM DIXIT DR M. S. KAMATH (*Alternate*)
Defence Food Research Laboratory, Mysore	DR G. K. SHARMA DR D. D. WADIKAR (*Alternate)*
Department of Chemical Technology, University of Calcutta	PROF ARUP MUKHERJEEE DR FAROOQUE ABDULLAH (*Alternate*)
Envirocare Labs Private Limited, Thane	DR PRITI AMRITKAR DR NILESH AMRITKAR (*Alternate*)
Food Ingredients Manufacturers & Suppliers of India Association	SHRI FIROZ H. NAQVI
Grasim Industry	SHRI PANKAJ KUMAR GUPTA
ICMR-National Institute of Nutrition, Hyderabad	DR J. PADMAJA
Indian Institute of Chemical Technology, Hyderabad	DR ASHOK KUMAR TIWARI DR T. KUMARAGURU (*Alternate*)
Indian Pharmacopoeia Commission, Ghaziabad	MS SHRUTI RASTOGI SHRI JAI SHIV CHAUHAN (*Alternate*)
Indian Salt Manufacturers Association, Ahmedabad	SHRI B. C. RAWAL SHRI P. R. DHRUVE (*Alternate)*

Organization	*Representative(s)*
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Office of the Salt Commissioner, Jaipur	SHRI M. A. ANSARI SHRI B. S. NAGAR (*Alternate*)
Protein Foods and Nutrition Development Association of India, Mumbai	SHRI BHUPINDER SINGH DR JAGADISH PAI (*Alternate*)
Roha Dyechem, Mumbai	SHRI ZAINULABIDIN DHANSE
VR Food Tech Pvt Ltd	DR ASHLESH PARCHURE DR N. RAM (*Alternate*)
BIS Directorate General	SHRIMATI SUNEETI TOTEJA SCIENTIST 'E'/DIRECTOR AND HEAD (FOOD AND AGRICULTURE DEPARTMENT) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE DEPARTMENT), BIS

(Continued from third cover)

The major changes brought out in this revision include the following changes:

a) Limit for chlorinated organic compound has been specified; and

b) Requirement of lead has been incorporated deleting the requirement of ‘Heavy Metals’.

The composition of the Committee responsible for the formulation of this standard is listed in Annex G

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rounding of numerical values (*second revision*)’. The number of significant places retained in he rounded off value should be the same as that of the specified value in this standard.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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